SYNTHESIS, CHARACTERIZATION AND FIRE RETARDANCY OF TITANIA-BASED MATERIALS COATED ON WOOD

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ABSTRACT

The number of fire cases in Malaysia has recently been increasing year after year, necessitating fire prevention measures. One prevention method is introducing fire retardant (FR) coating materials. FR coating material is a coating layer that works to prevent and reduce the probability of the material being flammable. Titania (TiO_2) nanoparticles (NPs) and silica/titania (SiO_2/TiO_2) nanocomposites (NCs) worked as the FRs coating materials in this project, while rubberwood functioned as the flammable substrate material. The fire performance behaviour was then investigated using cone calorimeter, thermogravimetric analyzer (TGA), and flammability tests. According to the findings, FR coating of TiO_2 NPs and SiO_2/TiO_2 (ratio of 0.1:1) NCs were able to increase the decomposition temperature (OD) of rubberwood by 41.32°C and 37.59°C, spontaneous ignition (SI) by 45.95°C and 32.6°C, and delayed ignition time (IT) by 79 s and 114 s. The results also proved the reduction in the intensity of fire (FI), heat release rate (HRR) by 43.32 kW/m^2 and 45.87 kW/m^2, rate of combustion (ROC) by 0.144 mm/s and 0.142 mm/s, mass loss rate (MLR) by 2.7 g/s and 2.9 g/s, and combustion efficiency (EHC) by 4.89 mJ/kg and 4.95 mJ/kg, respectively. The fuzzy logic system determined from these parameters that the HRR parameter should be considered as the key parameter that needs to be decreased in order to improve the FR performance of the TiO_2-based materials. The physicochemical properties of TiO_2-based materials coated on wood were then analyzed by various instruments and methods. Field emission scanning electron microscopy (FESEM) showed that the TiO_2-based materials coated on the wood are spherical in shape, in the nano-range (25 to 40 nm), and agglomerated on the surface of the rubberwood. Meanwhile, energy dispersive X-ray (EDX) confirmed that the presence of titanium (Ti) and silica (Si) as the primary elements. The principal functional groups of TiO_2 and SiO_2 were also visible by fourier transform infrared (FTIR) spectroscopy. The X-ray diffraction patterns (XRD) demonstrated that the TiO_2 in both TiO_2 and SiO_2/TiO_2 samples are present in the anatase phase with a lower crystallinity, corresponding to the bandgap energies (3.2 eV and 3.4 eV) determined using diffuse reflectance ultraviolet-visible spectroscopy (UV-Vis). Through the peel adhesion test, it was proven that the application of 3-aminopropyltrimethoxysilane (APTMS) as a surface modifying agent allowed strong adhesion between the TiO_2-based materials and wood and was uniformly coated. The wettability test showed that the surface of the rubberwood changed from superhydrophilic to hydrophilic. In conclusion, this study has demonstrated that TiO_2 and SiO_2/TiO_2 (0.1:1) are the best coating materials on wood that can successfully operate as fire retardant materials, displaying the potential to decrease the performance of wood burning.

ABSTRAK

Jumlah kes kebakaran di Malaysia sekarang ini telah meningkat dari tahun ke tahun, memerlukan langkah pencegahan kebakaran. Salah satu kaedah pencegahan adalah dengan memperkenalkan bahan salutan kalis api (FR). Bahan salutan FR adalah lapisan salutan yang berfungsi untuk mencegah dan mengurangkan keupayaan bahan menjadi mudah terbakar. Titanium (TiO_2) nanopartikel (NPs) dan silika/titanium (SiO_2/TiO_2) nanokomposit (NCs) berfungsi sebagai bahan salutan FR dalam projek ini, manakala kayu getah berfungsi sebagai bahan substrat yang mudah terbakar. Ujian ketahanan terhadap api diuji menggunakan peralatan kalorimeter kon, penganalisis termogravimetrik (TGA), dan ujian kebakaran. Menurut penemuan, salutan FR TiO_2 NPs dan SiO_2/TiO_2 NCs (nisbah 0.1:1) masing-masing mampu meningkatkan suhu penguraian (OD) kayu getah sebanyak 41.32°C dan 37.59°C, nyalaan spontan (SI) sebanyak 45.95°C dan 32.6°C, dan masa pencucuhan (IT) sebanyak 79 s dan 114 s. Hasil analisis juga membuktikan ianya mampu menurunkan keupayaan nyalaan api (FI), kadar pelepasan haba (HRR) sebanyak 43.32 kW/m^2 dan 45.87 kW/m^2, kadar pembakaran (ROC) sebanyak 0.144 mm/s dan 0.142 mm/s, kadar kehilangan jisim (MLR) sebanyak 2.7 g/s dan 2.9 g/s, dan kecekapan pembakaran (EHC) sebanyak 4.89 mJ/kg dan 4.95 mJ/kg. Sistem penentuan logikal mendapati bahawa parameter HRR harus diambil perhatian sebagai parameter yang perlu dikurangkan bagi memastikan prestasi TiO_2 sebagai bahan asas FR mampu bertindak dengan lebih cekap. Sifat fizikokimia bahan berasaskan TiO_2 yang dilapisi pada kayu kemudiannya dianalisa dengan pelbagai instrumen dan kaedah. Mikroskopi elektron pengimbas pelepasan medan (FESEM) menunjukkan bahawa bahan berasaskan TiO_2 yang dilapisi pada kayu berbentuk sfera, dalam julat nano (25 hingga 40 nm), dan melapisi permukaan kayu getah. Sementara itu, penyebaran tenaga sinar-X (EDX) mengesahkan kehadiran titanium (Ti) dan silika (Si) sebagai unsur utama. Kumpulan fungsi utama TiO_2 dan SiO_2 juga dapat dilihat menggunakan spektroskopi infra merah transformasi fourier (FTIR). Corak difraksi sinar-X (XRD) menunjukkan bahawa TiO_2 dalam kedua-dua sampel TiO_2 dan SiO_2/TiO_2 hadir dalam fasa anatase dengan kehabluran yang lebih rendah, sepadan dengan tenaga jurang jalur (3.2 eV dan 3.4 eV) yang ditentukan menggunakan spektroskopi pantulan resapan ultra lembayung nampak (UV-Vis). Melalui ujian pita pelekat, terbukti bahawa penggunaan 3-aminopropiltrimetoksisilana (APTMS) sebagai agen pengubah permukaan menghasilkan kelekatan permukaan yang lebih kuat antara bahan berasaskan TiO_2 dan kayu dan melapisi secara seragam. Ujian kelembapan menunjukkan bahawa permukaan kayu getah berubah dari superhidrofilik kepada hidrofilik. Kesimpulannya, kajian ini telah menunjukkan bahawa TiO_2 dan SiO_2/TiO_2 (0.1:1) adalah bahan salutan kalis kebakaran yang terbaik ke atas kayu, dengan menunjukkan potensi untuk menurunkan prestasi pembakaran kayu.

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LIST OF ABBREVIATIONS

ABS	-	Acrylonitrile butadiene styrene
APTMS	-	3-aminopropyltrimethoxysilane
ASTDR	-	Agency For Toxic Substances And Disease Registry
ASTM	-	American Society for Testing and Materials
BS	-	British Standard
BJC	-	builders' joinery and carpentry
DEFRA	-	Department for Environment, Food and Rural Affairs
DR UV-Vis	-	Diffuse reflectance ultraviolet-visible spectroscopy
DSM	-	Department of Standard Malaysia
DTI	-	Department of Trade and Industry
CB	-	Conduction band
EDX	-	Energy dispersive X-ray spectroscopy
EHC	-	Effective of Heat Combustion
EFRA	-	European Flame Retardant Association
EFSA	-	European Food Safety Authority
EPDM	-	Ethylene propylene diene methylene
FESEM	-	Field-emission scanning electron microscopy
FAO	-	Food and Agriculture Organization
FIRA	-	Furniture Industry Research Association
FPDSB	-	Forest Plantation Development Sdn. Bhd.
FRDM	-	Fire and Rescue Deparment of Malaysia
FR	-	Fire retardant
FRs	-	Fire retardants
FSA	-	Malaysia Fire Services Act
FTIR	-	Fire retardants
Gi-XRD	-	Grazing incidence X-ray diffraction
HRR	-	Heat release rate
IT	-	Ignition Time
JBPM	-	Jabatan Bomba dan Penyelamat Malaysia
LOI	-	Limiting oxygen index

MCA	-	Melamine cyanurate-fume
MDF	-	Medium density fiberboard
MOF	-	Malaysia of Financial
MLR	-	Mass loss rate
MPI	-	Ministry of Primary Industries
MPIC	-	Ministry of Plantation Industries and Commodities
MS	-	Malaysia Standard
MTC	-	Malaysian Timber Council
MTIB	-	Malaysia Timber Industry Board
MUF	-	Melamin urea formaldehyde
NCs	-	Nano-composites
NPs	-	Nano-particles
NFPA	-	National Fire Protection Association
PF	-	Phenol formaldehyde
PUSPEK	-	Pusat Penyelidikan Kebakaran
PVC	-	Polyvinyl chloride
ROC	-	Rate of combustion
TOF	-	Time of flame out
TTIP	-	Titanium (IV) isopropoxide
TGA	-	Thermal gravitional analyzer
UNHCR	-	United Nations High Commissioner for Refugees
UTM	-	Universiti Teknologi Malaysia
UV	-	Ultra violet
VB	-	Valence band
WCA	-	Water contact angle

LIST OF SYMBOLS

θ	-	Theta
%	-	Percent
t_s	-	Time in second
q	-	Heat release rate (kJ/s or kW)
Δh_c	-	Heat of combustion (kJ/g)
g	-	Gram
s	-	Second
m	-	Mass
Δh_{eff}	-	Effectiveness of heat combustion (mJ/kg)
m^n	-	Mass flux rate (kg/m^2s)
A	-	Area (m^2).
°C	-	Degree Celcius
ω	-	Omega
β	-	Betta
f	-	Coefficient
d	-	Distance
t	-	Time
ΔE	-	Energy
Δe^+	-	Energy release contribution
Δe^-	-	Energy loss
Σ	-	Sigma
F_n	-	Fuzzy number

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CHAPTER 1

INTRODUCTION

1.1 Research background

In Malaysia, there have been more than 45,000 building-related fire occurrences documented from 2010 to 2021 alone, averaging 7,292 events annually (FRDM, 2021). The number of these fire incidents keeps increasing yearly, which is a really alarming problem. These increases have a negative impact on the nation since they result in the loss of property and housing tax while also raising the government of Malaysia's service costs (MOF, 2018). The Malaysian government, in particular the Fire and Rescue Department of Malaysia (FRDM), is now working to raise public awareness of fire prevention through the organisation of numerous initiatives. But despite the steps taken to hold campaigns, the frequency of fires is still not going down. In addition to campaigns, better measures should be implemented by preventing uncontrollable combustion activity, awareness of the flammability materials, replacing the use of fire-friendly materials with non-fire-friendly materials, implementing non-flammability materials, or implementing a new technology based on the fire retardant concept.

Fire-friendly materials are present all around us, especially in homes, workplaces, and other environments are capable to produce danger to us, especially related to fires (Blye and Bacon, 1991). The fire-friendly materials are flammable substances that are frequently carbon-based and are referred to as fuel loads combined (Stellman, 1998). There are many different fuel loads accessible in various chemical and physical states (Martinez, 2007). These fuel loads may vary depending on how easily a fire starts, how quickly it spreads (flame speed), and how much power it can provide (rate of heat release) (Blye and Bacon, 1991). The idea of a fire tetrahedron, sometimes known as the fire triangle, is used to explain how flames start. It comprises of three basic parts: heat, fuel, and oxygen, which surround the fourth element, which is the chemical chain reactions that occur during combustion activities (Friedman, 1998). When all of these components are connected and mixed, fires are started (Daeid, 2004).

Typically, materials can have a layer of coated film applied to their surface; the majority of these coated films are composed of traditional organic components (Baghdachi, 2011). It is well-recognised that organic materials make excellent fuel since they are simpler to ignite, melt, and drip. When they are exposed to heat or fire, this could result in serious injuries and destruction (Goldsmith, 2011). FRs have developed into a chemical class that consistently draws significant scientific and public attention. An example of organic material is wood. Wood is a porous material that has fibrous structural tissues, is classified as an organic material, has rich with a natural composite of cellulose fibers, provides strong tension, and is embedded in a matrix of lignin (Panda, 2016). Wood is one of the most commonly used materials, and almost any type of wood can be used to build furniture (Pařil, 2016). Each type of wood has its own unique characteristics, which in turn can add different degrees of warmth, emphasis, and beauty, to its surrounding decor. Even though it is one of the most commonly used materials, as an organic material, wood can be easily exposed and turned into fuels, which are easily burned when exposed to fire. Therefore, the application of wood in the furniture and decoration industry needs extra protection to reduce the risk of degradation and ignition by fire.

One of the best ways to reduce the possibility of a fire involving flammable or combustible materials is by introducing reactive or additive fire retardant (FR) materials (Betts, 2008). FR materials have developed into a chemical class that consistently draws significant scientific and public attention. Every production of materials based on wood, plastic, or textile must take into account the level of combustibility of the product. In order to reduce the level of combustibility product, the reactive or additive fire retardant materials need to be prepared together as a way to ensure the product produced has a lower combustibility level (Innes, 2011). The reactive FR materials, for example, by reducing the particle size to nano-size, the effectiveness of FR material becomes better. Smaller particles size of FR materials will give them a larger area to cover the surface of the substrate. According to Wang *et al*. (2007), the smaller particle size produced will provide a better coating layer and at the same time will decrease the combustibility of the substrate.

In recent years, nanoparticles (NPs) applications in the coatings industry have grown significantly. This increase is caused by the physicochemical characteristics of NPs, which have smaller particle sizes between 0.1 and 100 nm. In reality, several performance issues brought on by some pricey products in the coating industry have been addressed using the potential of NPs (Fernando and Sung, 2009; Thirumal, 2017). This is because NPs, which have a variety of benefits and potential for the coatings business, look good and are useful (Li *et al.*, 2016; Khanna, 2008). Additionally, NPs coating materials can increase opacity, foster better surface-coating interaction, increase durability against combustible and flammable materials, and enhance the mechanical, thermal, and electrical properties of the substrates (Small *et al.*, 2006; Arao, 2015). A good example of NPs would be titania (TiO_2), where the fabrication of TiO_2 using various methods has been carried out in order to increase the efficiency of TiO_2 as a non-combustible filler (Tayeb and Hussein, 2015).

TiO_2 NPs have been used as an inorganic FR and for hydrophilicity reasons, which have both brought attention to their outstanding qualities. In contrast to other conventional FR materials like halogenated, phosphorus, and nitrogen, which are most likely to become toxic to the environment over time, TiO_2 NPs have a strong oxidation power, are non-toxic, environmentally friendly, have high photostability, chemical stability, mechanical hardness, less moisture absorption, and have high thermal stability (Chae, 2003; Sun *et al*., 2012). The high oxidation properties of TiO_2 NPs in fire protection coatings are due to the formation of nanoscale ceramic-like protective materials in char structures (Wang *et al.*, 2006). Of late, TiO_2 NPs have been popularly used as a paint additive in coating industries due to the white pigments' appearance, high brightness, and a very high refractive index (Chemours, 2019). These appearances have unique characteristics in terms of band position and surface structure (Chae, 2003).

Although TiO_2 NPs are able to improve the thermal behaviour, mechanical, and flame retardancy of materials, the presence of other fire retardants is needed to give extra synergy effect for fire retardancy purpose (Arao, 2015). According to Sun *et al*. (2012), the combination of dual inorganic materials, such as titania/zinc oxide (TiO_2/ZnO) or titania/silicon dioxide (TiO_2/SiO_2) composites, when coated on easily combustible materials, such as wood, showed significant improvements in fire resistivity.

Despite all the studies on wood that have been reported for the development of FR materials, there are still several issues that need to be addressed and renewed. Sun *et al*. (2013) and Rosales *et al*. (2018) targeted TiO_2 NPs as material to improve wood's fire resistivity, durability, and to increase the wood hardness properties. According to Yew *et al.* (2015), the selection of types of FR material is very important especially to ensure the effectiveness and durability of the material against fire resistance. Therefore, the use of TiO_2 NPs as a fire protection coating for wood should be further investigated. Other methods such as combining two or more materials with TiO_2 are also very influential and able to increase the fire resistance of the material, as well as increase the resistance of the substrate (Yew *et al*., 2015). Phosphoric acid, boric acid, ZnO, 3-aminopropyltrimethoxysilane (APTMS), and SiO_2 nanostructures are suitable examples to be used as additives or coupling agents in order to increase the effectiveness to protect the substrate and increase the interfacial strength, especially for wood-based composites (Hübert & Mahr, 2017). The function of this protective layer is mainly to increase the mechanical resistance, moisture and thermal properties of the wood materials (Zhang *et al*., 2019).

In this work, the preparation of size-controlled TiO_2 NPs by sol-gel method with TTIP as the Ti source, combined with SiO_2 as additional material, to increase the fire retardancy of the coated woods, have been carried out. Prior to coating, the woods underwent pre-treatment using 3-aminopropyltrimethoxysilane (APTMS) as the coupling agent to improve the adhesion of TiO_2-based materials on the surface of the woods (Then *et al*., 2015). The synthesized materials were then coated onto wood through the dip-coating method before being characterized by various methods to determine the physicochemical properties. The coated woods then underwent fire retardancy testing and measurement of the water contact angle (WCA) to study the interaction between the coating surface with water. The fuzzy logic system was then used to determine the dominant factor of fire retardant coating used. The illustration of the concept of this study is shown in Appendix A.

1.2 Problem statement

The number of fire incidents in Malaysia has recently been worryingly rising and has now reached an extremely concerning level, particularly for materials that are combustible and flammable and are frequently used in our daily lives, particularly for materials made of wood, such as furniture and home appliances. Therefore, significant research has been done to create an effective protection layer to cover the flammable and combustible materials to prevent them from rapidly igniting in order to overcome this drawback. One of the solutions is by providing FR coating materials, which can increase the fire resistivity of the coated materials. Unfortunately, FR materials that have been produced in Malaysia are relatively limited as most individuals, including manufacturers, suppliers, dealers, and customers, are not interested due to the lack of information and awareness, as well as the higher cost of production. Furthermore, in the Malaysia Standard (MS) for fire safety and protection, the standards of fire protection for furniture manufacturing have not been mentioned as one of the requirements (DSM, 2020). Almost all the FR materials available are imported from other countries.

The purpose of the application of the coating on the surface of materials is not only for aesthetic reasons but can also enhance scratch resistance, adhesion resistance, UV resistance, fire resistance, moisture resistance, anti-microbial activity, self-cleaning activity and also to add gas barrier properties to the substrate (Tri, Rtimi, & Ouellet-Plamondon, 2019; Fernando and Sung, 2009). According to Khare *et al.* (2015), the application of coating onto the surface of the material can be used to stabilize the particles, agglomeration and withstand dissolution, and discharge noxious ions. Nevertheless, most of the FR materials are made up of halogenated-based and phosphorous-based substances which can present adverse impacts on the environment and can be highly toxic when burned. Hence, many countries no longer use these FRs (Babrauskas *et al*., 2014). Meanwhile, it was found that nitrogen-based FRs showed many advantages, such as low toxicity and being environmentally friendly. However, the amount of nitrogen FRs is needed in large quantities to increase the effectiveness of fire retardancy (Horacek & Grabner, 1996).

In order to solve these problems, an approach that uses an inorganic material such as metal oxide nanocomposites (NCs) to obtain a good coating quality of FRs coating has been undertaken. According to Tri *et al*. (2019) and Zanatta *et al*. (2017), metal oxides coatings, such as TiO_2 NPs and SiO_2 NPs, are suitable to use in FRs applications due to good passivation, stability at high temperatures, chemical stability, mechanical hardness and smooth for coating processes. Out of all these, TiO_2 and SiO_2 stand out due to their higher mechanical durability and higher temperature stability. Application of both metal oxides to the wood will increase the wood's hardness and is also capable of covering the wood's pores and poor adsorbent by reducing water interaction to the surface of the coated wood (Xuan *et al.*, 2018). To support the use of TiO_2 and SiO_2 as FR coating materials in improving the fire retardancy properties of wood, the fuzzy logic system has been used to justify the usage.

1.3 Objectives of the study

The objectives of this study are:

i. To synthesize TiO_2 NPs-based materials (TiO_2 NPs and SiO_2/TiO_2 NCs) as FRs coating on wood
ii. To study the ability of the TiO_2 NPs-based materials coated on wood as FRs through a fire performance test.
iii. To study the dominant factor of FRs coating by the fuzzy logic system.
iv. To determine the physicochemical properties of the TiO_2 NPs-based materials coated on wood.

1.4 Scope of study

This research focuses on using TiO_2 NPs as a based FR coating materials and SiO_2 NPs as additive materials for TiO_2-based coating materials, which are used as a fire shield material on the surface of the wood. The effectiveness of additive materials of SiO_2 was also investigated. Following that, the dominant factor of the coating was determined using the fuzzy logic system. The TiO_2 NPs-based materials have been synthesized using the sol-gel method at a lower temperature (90 °C). The different compositions of SiO_2 have also been prepared with the molar composition ratio between SiO_2 and TiO_2 being 0.05:1, 0.1:1 and 0.5:1, respectively. Rubberwood with sizes of 100 mm x 100 mm x 5 mm and 130 mm x 5 mm x 5 mm was chosen as a coating substrate due to its popularity as furniture-based material and it is also in a class of combustible materials (Ratnasingam *et al*., 2019; Ratnasingam, 2017). The coating process was carried out utilising the dip-coating method at room temperature and dried overnight at a temperature of 130 °C.

Meanwhile, the flammability test of the coated woods has been carried out by following the British Standard of fire tests on building materials and structures and America Society for Testing and Materials, E1354 (BS 476, 1989; 1997; ASTM 1999). The test was conducted by exposing the coated wood's surface to a flame source from a flame burner and then analysing the results using a cone calorimeter analyzer and thermogravimetric analyzer. The fire performance test was carried out at the Fire Forensic Laboratory of FRDM, Johor state and JBPM Fire Research Centre (PUSPEK), Nilai, Negeri Sembilan

The fuzzy logic system was then utilised to determine the dominant factor in enhancing the fire retardancy qualities of the TiO_2 NPs-based coating materials in order to anticipate the dominant factor of the TiO_2 NPs-based coating materials on wood as FR. The combination of the different factors of the coating, such as heat release rate (HRR), rate of combustibility (ROC), mass loss rate (MLR) and ignition time (IT), was elucidated using the fuzzy graph. Specifically, the fuzzy logic system includes four main stages, which are (1) data collection, (2) development of fuzzy logic controller, (3) construction of fuzzy inference system and (4) assessment of the result by sensitivity analysis.

Lastly, the determination of the physicochemical properties of the coated wood was analyzed by several instruments. The surface morphology and measurement of the particle size were done using field-emission scanning electron microscopy with energy-dispersive X-ray spectroscopy (FESEM-EDX). The determination of the functional groups was carried out using Fourier transform infrared (FTIR) spectroscopy, crystallinity study by using X-ray diffraction (XRD), and bandgap energy determination by using diffuse reflectance ultraviolet-visible spectroscopy (DR UV-vis), peel adhesion test and the properties of water spreads on the surface of coated wood were evaluated through the measurement of WCA by measuring the contact angle (θ) of a water droplet.

1.5 Significance of the study

This study aims to utilize TiO_2-based coating materials, with TiO_2 NPs as the main element and SiO_2 NPs as the additional element, in the development of the FRs coating materials. It is expected that the TiO_2-based materials coated on the wood will be able to reduce the combustibility of the wood substrate, extend the duration of the ignition time, reduce the rate of fire spread, and be capable of self-extinguisher the fire. Next, in order to determine the factor that helps or interferes with the effectiveness of TiO_2-based coating materials as FR materials, the fuzzy logic system was used to determine the dominant factor involved. Apart from that, with the presence of TiO_2 NPs and SiO_2 NPs as non-flammable materials in the coating industry, it is hoped that such an attempt would contribute to the improvement the fire safety, extend the duration of the ignition time, reduce the rate of fire spread, hinder the combustion process, and ultimately reduce the number of fire cases in Malaysia.

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LIST OF PUBLICATIONS

NO.	TITLE	PUBLISHER	YEAR
1	Fire-Retardancy Of Wood Coated By Titania Nanoparticles	AIP Concerence Proceeding 2155	2019